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SOURCE Sintezy Organicheskikh Soyedineniy, Sbornik II (Syntheses of Organic
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CHLOROMETHYLPHOSPHONIC ACID

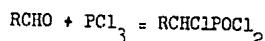
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Chloromethylphosphonic acid is prepared on the basis of the following reactions:
 $PCl_3 + CH_2O \rightarrow ClCH_2POCl_2 \rightarrow 2ClCH_2PO(OH)_2$

Chloromethylphosphonic acid was first obtained by A.Ya. Yakubovich, V.A. Ginsburg, and S.P. Makarov (1) from phosphorus trichloride and diazomethane with subsequent chlorination, treatment with sulfur dioxide, and hydrolysis.

Later it was synthesized by hydrolyzing its acid chloride, which was obtained by the action of phosphorus pentachloride on hydroxymethylphosphonic acid. This, in turn, was prepared from formaldehyde and phosphorus trichloride (2). Neither of these methods furnishes a good procedure for the preparation of chloromethylphosphonic acid.

The present method of obtaining chloromethylphosphonic acid (3) is based on the reaction of aldehydes with phosphorus trichloride taking place at 180-250° according to the following scheme (4):



The acid chloride of chloromethylphosphonic acid gives a 60-65% yield and readily converts to chloromethylphosphonic acid on being reacted with water.

Description of the Synthesis

1. The Acid Chloride of Chloromethylphosphonic Acid

Two hundred g of phosphorus trichloride and 30 g of paraformaldehyde, which has been carefully dried and ground to a fine powder, are heated in a one-liter autoclave to 250° for a period of 10 hours. The reaction mixture is

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fractionated twice under vacuum. The acid chloride of chloromethylphosphonic acid is collected at 87-88° and 15 mm. 100-108 g are obtained (60-64% of the theoretical yield).

2. Chloromethylphosphonic Acid

The acid chloride of chloromethylphosphonic acid hydrolyzes with heating after addition of one part of the acid chloride to 15 parts of water. When a large quantity is hydrolyzed, external cooling is necessary. The solution that forms is evaporated in a dish over a water bath. The hydrogen chloride is removed from the remaining sirup by evaporation with water and the product is then placed in a desiccator charged with caustic. The substance crystallizes on standing. It is recrystallized by dissolving it in a mixture of ether and toluene (1 : 1) and leaving in a desiccator containing sulfuric acid and paraffin for the gradual evaporation of the solvent.

Properties

The acid chloride of chloromethylphosphonic acid is a colorless liquid that smokes in the air. It is insoluble in water and hydrolyzes rapidly. Its boiling point is 87-88° at 15 mm; 52-53° at 2 mm; n_D^{20} 1.4978; d_4^{20} 1.6361.

The crystals of chloromethylphosphonic acid melt at 86-87.5°. The crystals are soluble in water, alcohol, and glacial acetic acid; but insoluble in benzene, petroleum ether, and dichlorethane.

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